

Table I. Crystal Data and Atomic Parameters for (2,2,2-crypt-K⁺)₂HgTe₂²⁻·en

monoclinic, $P\bar{1}$; $a = 11.263$ (1) Å, $b = 12.171$ (2) Å, $c = 10.335$ (2) Å, $\alpha = 107.00$ (2)^o,
 $\beta = 91.18$ (2)^o, $\gamma = 88.36$ (2)^o; $V = 1352.2$ (3) Å³, $Z = 1$, $fw = 1347.09$, $\rho_{\text{calcd}} = 1.65$ g/cm³

atom	x	y	z	β_{11} ^a	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Hg	0.0	0.0	0.0	7.50 (3)	7.16 (3)	9.93 (5)	-0.14 (2)	-0.10 (3)	2.03 (3)
Te	0.15181 (5)	0.16038 (5)	0.09552 (6)	10.73 (6)	9.28 (5)	15.78 (8)	-2.85 (4)	-0.27 (5)	1.70 (5)
K	0.6173 (1)	0.2693 (1)	0.6033 (1)	6.3 (1)	6.6 (1)	9.1 (1)	-0.3 (1)	0.1 (1)	1.8 (1)
N(1)	0.8540 (6)	0.2251 (6)	0.4741 (8)	7.7 (5)	11.8 (7)	17.8 (10)	0.0 (5)	2.4 (6)	2.5 (6)
N(2)	0.3780 (5)	0.3160 (6)	0.7320 (6)	8.4 (5)	10.3 (6)	12.7 (7)	0.0 (4)	2.6 (5)	2.4 (5)
O(1)	0.6392 (5)	0.1013 (5)	0.3458 (5)	11.1 (5)	11.8 (5)	10.4 (6)	0.7 (4)	1.3 (4)	1.2 (4)
O(2)	0.4143 (4)	0.1802 (4)	0.4526 (5)	8.5 (4)	8.5 (4)	15.1 (7)	-1.8 (3)	-2.0 (4)	0.2 (4)
O(3)	0.8108 (5)	0.2118 (5)	0.7430 (7)	10.8 (5)	12.7 (6)	19.1 (9)	1.2 (4)	-3.0 (5)	6.6 (6)
O(4)	0.5776 (5)	0.2079 (5)	0.8403 (5)	12.7 (6)	12.5 (5)	11.0 (6)	-0.6 (4)	-1.6 (5)	3.9 (5)
O(5)	0.7399 (5)	0.4542 (4)	0.5584 (6)	9.5 (5)	8.2 (4)	16.4 (7)	-2.8 (3)	1.4 (5)	1.1 (4)
O(6)	0.5417 (5)	0.4998 (4)	0.7343 (6)	12.3 (5)	7.5 (4)	15.3 (7)	0.7 (4)	0.6 (5)	3.5 (4)

atom	x	y	z	B, Å ²	atom	x	y	z	B, Å ²
C(1)	0.850 (1)	0.114 (1)	0.366 (1)	7.0 (2)	C(11)	0.469 (1)	0.248 (1)	0.915 (1)	6.5 (2)
C(2)	0.745 (1)	0.101 (1)	0.272 (1)	7.1 (2)	C(12)	0.367 (1)	0.241 (1)	0.818 (1)	6.2 (2)
C(3)	0.536 (1)	0.092 (1)	0.263 (1)	6.5 (2)	C(13)	0.877 (1)	0.319 (1)	0.418 (1)	7.0 (2)
C(4)	0.428 (1)	0.076 (1)	0.341 (1)	6.3 (2)	C(14)	0.862 (1)	0.438 (1)	0.519 (1)	7.0 (2)
C(5)	0.305 (1)	0.178 (1)	0.518 (1)	6.4 (2)	C(15)	0.727 (1)	0.556 (1)	0.670 (1)	6.5 (2)
C(6)	0.286 (1)	0.291 (1)	0.625 (1)	6.1 (2)	C(16)	0.600 (1)	0.588 (1)	0.690 (1)	6.3 (2)
C(7)	0.946 (1)	0.216 (1)	0.574 (1)	7.5 (2)	C(17)	0.417 (1)	0.520 (1)	0.745 (1)	5.8 (2)
C(8)	0.910 (1)	0.152 (1)	0.669 (1)	7.4 (2)	C(18)	0.365 (1)	0.441 (1)	0.812 (1)	6.1 (2)
C(9)	0.784 (1)	0.167 (1)	0.855 (1)	7.1 (2)	N(en)	0.954 (1)	0.440 (1)	0.135 (2)	14.8 (5)
C(10)	0.679 (1)	0.229 (1)	0.928 (1)	7.0 (2)	C(en)	0.977 (4)	0.527 (3)	0.047 (4)	22.0 (12)

^a Thermal parameters ($\times 10^3$) of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$ are listed.

The K-N distances are 2.970 (7) and 2.996 (6) Å, and the K-O distances vary from 2.774 (6) to 2.853 (6) Å. There is a possible shift (0.026 Å, 2.8 σ) of the potassium atom toward the end of the crypt ligand associated to a greater extent with the anion and en molecule. Similar effects have been observed in the structures of the 2,2,2-crypt-potassium salts of Te₃²⁻ (0.09 Å, 2.3 σ) and Bi₄²⁻ (0.09 Å, 3 σ).^{2,8} We also note some small but significant differences among the K-O distances.

The HgTe₂²⁻ anion, which must be linear by virtue of the inversion center, has a Hg-Te bond length of 2.5890 (8) Å. This may be compared with a calculated value of 2.65 Å based on covalent radii for digonal mercury (1.30 Å)⁹ and tellurium (1.35 Å).¹⁰ A slightly closer value (2.63 Å) may be obtained by using a covalent radius for mercury calculated from the Hg-Hg distances in Hg₃(AsF₆)₂ and Hg₃(AlCl₄)₂.^{11,12} In either case the observed distance appears significantly smaller than calculated. Comparison with the Hg-Te distance (2.78 Å)¹³ in HgTe, which has the zinc-blende (sphalerite) structure, is not appropriate here as both atoms are tetrahedrally bonded in this material.

The HgTe₂²⁻ anion has an analogue with the lighter group 6 element oxygen in HgO₂²⁻, which has been structurally characterized in the compounds Na₂HgO₂ and K₂HgO₂.¹⁴ In both, the HgO₂²⁻ group is linear, and the Hg-O distances are 1.96 Å ($\times 2$) and 1.93 Å ($\times 2$), respectively. Here we note that the Hg-O distance is significantly less than the sum of the covalent radii for the respective elements (2.03 Å), as is also observed for HgTe₂²⁻. For cinnabar, HgS, there are helical chains in which the Hg has two nearest sulfur neighbors at 2.36 Å, two more at 3.10 Å, and two more at 3.30 Å, with a S-Hg-S bond angle of 172^o.¹⁵ In this case the Hg-S distance appears greater than the sum of the respective co-

valent radii (2.32 Å), presumably as a result of the extra Hg-S contacts, which reduce the bond order within the chain and increase the directly bonded Hg-S distance. Work on the mercury-tin system is continuing.

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Registry No. (2,2,2-crypt-K⁺)₂HgTe₂²⁻, 79172-66-0; KHgTe, 79172-67-1.

Supplementary Material Available: Listings of bond distances and angles in the cations and en and structure factor amplitudes, Tables II and III (14 pages). Ordering information is given on any current masthead page.

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Crystal and Molecular Structure of Trichloro[1,3-dimethyl-2(3H)-imidazolethione]antimony(III)

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In the structure of AsCl₃dmit^{2a} (dmit = 1,3-dimethyl-2-(3H)-imidazolethione) and the structure of AsCl₃NMe₃,^{2b} notable structural differences were observed. Both complexes

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could be described as pseudo trigonal bipyramidal (ψ -TBP) with the main differences being the relative position of the ligand and the degree of intermolecular interaction in the solid state. $\text{AsCl}_3\text{NMe}_3$ is monomeric, and the amine occupies an axial position in the ψ -TBP configuration. AsCl_3dmit is dimeric linked by long $\text{As}\cdots\text{Cl}$ bridges to the neighboring molecule related by an inversion center. The ligand (dmit) occupies an equatorial rather than an axial position, while the chlorines occupy two axial positions as well as the remaining equatorial site. Since the chlorine bridge bonds trans to the equatorial chlorine atom, when the bridging atoms are included, the coordination geometry of AsCl_3dmit can best be described as two distorted square pyramids sharing a common edge.

The structure reported for the aniline (anil) adduct of SbCl_3 is similar to the structure described for $\text{AsCl}_3\text{NMe}_3$.³ The SbCl_3anil structure is also ψ -TBP with the amine in an axial position. No intermolecular associations outside of van der Waals type appear in the solid state. For this reason the crystal and molecular structure study of SbCl_3dmit , described below, was undertaken, and the results were compared to the structures noted above.

Crystallographic Section

The synthesis and initial characterization of SbCl_3dmit have been reported. A yellow crystal of this compound with approximate dimensions $0.3 \times 0.3 \times 0.4$ mm was mounted on a glass fiber with epoxy cement such that the longest crystal dimension was approximately parallel to the fiber axis.

Unit cell parameters and the orientation matrix were determined on a Syntex P2₁ four-circle diffractometer equipped with a graphite monochromator (Bragg 2θ angle = 12.2°) using $\text{Mo K}\alpha$ radiation at a takeoff angle of 6.75° . Fifteen reflections were machine centered and used in least-squares refinement of the lattice parameters and orientation matrix. Unit cell parameters obtained were $a = 12.179$ (6) Å, $b = 7.523$ (5) Å, $c = 12.306$ (6) Å, $\beta = 98.82$ (5) $^\circ$, and $V = 1114$ Å³. The calculated density of 2.12 g cm⁻³ for 4 formula units per unit cell agrees well with the experimental density of 2.08 g cm⁻³ measured by the flotation method using a mixture of chloroform and 1,1,2,2-tetrabromoethane. ω scans of several low- 2θ -angle reflections gave peak widths at half-height of less than 0.10° , indicating a satisfactory mosaic spread for the crystal.

Intensity data for zero and upper levels were collected at a rapid scan rate and the intensities examined carefully for systematic absences. The absence of $(0k0)$, $k = \text{odd}$, and $(h0l)$, $l = \text{odd}$, is consistent with only space group $P2_1/c$ (No. 14).⁶

Intensity data were collected with use of θ - 2θ scans with X-ray source and monochromator settings identical with those used for determination of the unit cell parameters. A variable scan rate of 29.3 – 4.88° min⁻¹ was used, and a scan width of 2.0° was sufficient to collect all of the peak intensity. Stationary background counts were measured at the beginning (bgd1) and at the end (bgd2) of each scan with a total background to scan time ratio, TR, of 1.0. No significant fluctuations were observed in the intensities of three standard reflections $[(0,0,10)$, $(10,0,0)$, $(060)]$ monitored every 97 reflections. Intensities were calculated from the total scan count (CT) and background counts by the relationship $I = \text{CT} - (\text{TR})(\text{bgd1} + \text{bgd2})$. The intensities were assigned standard deviations according to the formula $\sigma(I) = [\text{CT} + (\text{TR})^2(\text{bgd1} + \text{bgd2})]$ from a total of 2235 reflections collected in a complete quadrant of data out to $2\theta = 50^\circ$; 1800 were accepted as statistically above background on the basis that $F > \sigma(F)$. Lorentz and polarization corrections were made in the usual way. No corrections for absorption were applied.

Structure Solution and Refinement

Computations were performed with use of SHELX-76⁷ and ORTEP⁸

Table I. Final Positional Parameters

	x/a^a	y/b	z/c
Sb	0.3426 (3)	0.2442 (4)	0.1973 (3)
S	0.3296 (1)	0.1933 (2)	0.3965 (1)
Cl1	0.1704 (1)	0.0586 (2)	0.1377 (1)
Cl2	0.2303 (1)	0.5127 (2)	0.1841 (1)
Cl3	0.5107 (1)	0.4275 (2)	0.3027 (2)
N1	0.1177 (4)	0.0617 (6)	0.3885 (3)
N2	0.2447 (4)	-0.1411 (7)	0.4162 (4)
C1	0.2273 (4)	0.0318 (7)	0.3984 (4)
C2	0.0623 (8)	0.2325 (9)	0.3673 (9)
C3	0.0636 (6)	-0.0974 (8)	0.3985 (5)
C4	0.1444 (8)	-0.2208 (10)	0.4152 (7)
C5	0.3564 (9)	-0.2317 (12)	0.4370 (9)
H2	-0.0219 (8)	0.1984 (9)	0.3765 (9)
H2'	0.0662 (8)	0.2602 (9)	0.2819 (9)
H2''	0.0881 (8)	0.3487 (9)	0.4162 (9)
H3	-0.0195 (6)	-0.0961 (8)	0.4190 (5)
H4	0.1585 (8)	-0.3612 (10)	0.4033 (7)
H5	0.3503 (9)	-0.3719 (12)	0.4540 (9)
H5'	0.4045 (9)	-0.1667 (12)	0.5070 (9)
H5''	0.3967 (9)	-0.2137 (12)	0.3656 (9)

^a Estimated standard deviations in the least significant figures are given in this and subsequent tables.

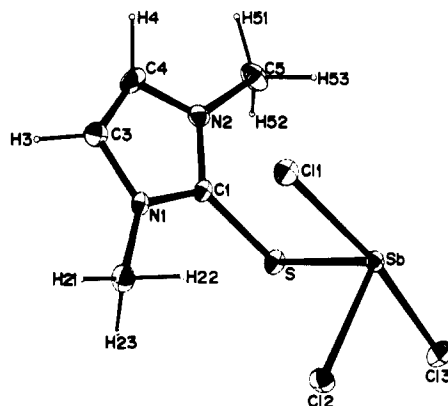


Figure 1. Molecular architecture of the title compound showing the thermal ellipsoids at the 50% probability level.

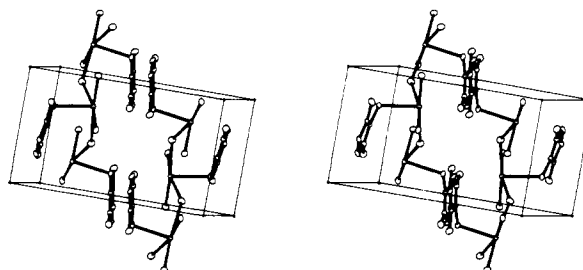


Figure 2. Stereodrawing showing the packing of molecules in the unit cell. In the drawing the a axis is approximately horizontal and b is vertical.

on the Univac 90/80 computer. For structure factor calculations, the scattering factors were taken from Cromer and Waber's tabulation.⁹ The scattering factors for all atoms except hydrogen were corrected for the real and imaginary anomalous dispersion components.⁹ The agreement factors were defined in the usual way as

$$R = (\sum ||F_o| - |F_c||) / (\sum |F_o|)$$

$$R_w = [\sum (|F_o| - |F_c|)(w^{1/2}) / \sum (|F_o|)(w^{1/2})]$$

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Table II. Thermal Parameters^a

	U_{11}^b	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sb	0.0305 (5)	0.0301 (5)	0.0217 (5)	0.0027 (1)	0.0069 (3)	0.0009 (1)
S	0.0373 (9)	0.0430 (9)	0.0205 (8)	-0.0001 (6)	0.0039 (6)	-0.0092 (7)
Cl1	0.0510 (10)	0.0519 (10)	0.0281 (8)	-0.0073 (7)	0.0040 (6)	-0.0104 (7)
Cl2	0.0427 (10)	0.0389 (9)	0.0458 (9)	0.0068 (6)	0.0042 (7)	0.0110 (6)
Cl3	0.0430 (9)	0.0459 (9)	0.0568 (10)	0.0042 (7)	0.0046 (7)	-0.0114 (6)
N1	0.0267 (21)	0.0383 (25)	0.0143 (21)	0.0021 (17)	0.0031 (15)	-0.0037 (18)
N2	0.0467 (29)	0.0330 (24)	0.0285 (25)	-0.0006 (20)	0.0114 (20)	0.0014 (20)
C1	0.0292 (25)	0.0346 (27)	0.0169 (23)	-0.0003 (20)	0.0027 (19)	-0.0074 (20)
C2	0.0360 (45)	0.0496 (45)	0.0523 (56)	0.0158 (30)	0.0089 (38)	0.0077 (27)
C3	0.0580 (37)	0.0384 (32)	0.0240 (28)	0.0068 (24)	0.0108 (26)	0.0023 (38)
C4	0.0715 (53)	0.0407 (35)	0.0311 (40)	0.0117 (29)	0.0133 (35)	-0.0138 (33)
C5	0.0524	0.0558 (55)	0.0450 (57)	0.0026 (30)	0.0058 (47)	0.0186 (30)

^a The form of the anisotropic thermal ellipsoids is $\exp[-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl)]$.
^b U for all H atoms is 0.075 (11) Å².

Table III. Selected Bond Distances (Å) and Angles (Deg)

Sb-S	2.511 (1)	C2-N1	1.456 (9)
Sb-Cl1	2.532 (2)	C3-N1	1.381 (7)
Sb-Cl2	2.431 (1)	C1-N2	1.330 (7)
Sb-Cl3	2.636 (1)	C4-N2	1.360 (10)
C1-S	1.743 (5)	C5-N2	1.508 (10)
C1-N1	1.341 (7)	C3-C4	1.348 (11)
Cl1-Sb-S	91.3 (1)	C1-N2-C4	108.2 (6)
Cl2-Sb-S	94.1 (1)	N2-C4-C3	109.2 (6)
Cl3-Sb-S	76.2 (1)	C4-C3-N1	105.4 (6)
Cl2-Sb-Cl1	90.4 (1)	C3-N1-C1	108.8 (5)
Cl3-Sb-Cl1	167.4 (1)	N1-C1-S	125.8 (4)
Cl3-Sb-Cl2	89.1 (1)	N2-C1-S	126.0 (4)
N1-C1-N2	108.0 (4)		

In all least-squares refinements, the quantity minimized was $\sum(|F_o| - |F_c|)^2$. A weighting scheme based on counting statistics ($w = 1/[(\sigma(F))^2 + 0.05F^2]$) was employed for calculating R_w and in least-squares refinement. The refinement converged with $R = 0.064$ and $R_w = 0.075$. A listing of the observed and calculated structure amplitudes is available.¹⁰

Final positional parameters and thermal parameters are given in Tables I and II. Selected bond distances and angles are shown in Table III.

Discussion

Figure 1 shows the molecular structure with the atoms as they are identified in the tables. Figure 2 shows the local geometry about the antimony atom and the packing of the molecules in the unit cell. There are two possible ways of viewing the structure. Considering only the geometry of the bonded atoms comparison can be made to the structure of SbCl_3anil and AsCl_3dmit . From this point of view the geometry resembles that of the ψ -TBP description of AsCl_3dmit with the ligand in an equatorial position and a near linear Cl-Sb-Cl group (167.4°, Table III).

The total bonding picture for SbCl_3dmit , however, is more complex than that described by a simple ψ -TBP unit. As Figure 2 shows, there are two intermolecular contacts from neighboring molecules that are within the van der Waals radii of the atoms involved. Thus the total coordination geometry is more accurately described as a distorted octahedron. The bridging chlorine, Cl(3)', comes from the neighboring molecule related by a screw axis while the sulfur, S'', comes from a glide-plane related molecule. The Sb...Cl(3)' bridge distance of 2.98 Å indicates a relatively strong interaction compared to the van der Waals sum of 4.0 Å for these atoms.¹¹ The Sb...S'' bridge of 3.76 Å is apparently less intense since the van der Waals estimate for these atoms is also ca. 4.0 Å.¹¹ The combination of these two intermolecular contacts result in

polymeric zig-zag double sheets of $\text{SbCl}_4(\text{dmit})_2$ octahedra linked in one direction by chlorine atom bridges displaced cis to each in the coordination sphere of each antimony atom and in the other direction by sulfur bridges displaced roughly trans to each other about each antimony atom.

The observed bond distances in SbCl_3dmit compared to those observed in SbCl_3anil may be rationalized on the basis of intermolecular contacts. The Sb-Cl bond distances for SbCl_3dmit are on the average much longer than those observed for SbCl_3anil .³ The longest Sb-Cl bond in SbCl_3anil is (as expected) the Sb-Cl(axial) bond at 2.516 (16) Å. The two axial Sb-Cl bonds in SbCl_3dmit are longer at 2.523 (2) and 2.636 (1) Å for Sb-Cl(1) and Sb-Cl(3), respectively. As in the case of AsCl_3dmit , the unequal Sb-Cl axial bond lengths may be rationalized by the fact that the longer distance is associated with the atom involved in bridging. A similar effect is seen in the dimeric structure of SeBr_2tmtu (tmtu = tetramethylthiourea).¹²

The "equatorial" Sb-Cl(2) bond distance of 2.431 (1) Å in SbCl_3dmit is also longer than either of the two corresponding bonds in SbCl_3anil (2.335 (18) and 2.323 (14) Å). The bridging Cl(3)' atom is displaced trans to Cl(2) in the coordination sphere of antimony, and thus the longer bond in SbCl_3dmit may be rationalized on the basis of a trans bond lengthening effect¹³ arising from close intermolecular contacts. The same reasoning may be used to explain the longer Sb-S distance of 2.511 (1) Å in SbCl_3dmit as compared to the sum of the covalent radii for these atoms (2.44 Å),¹¹ since the bridging S'' atom is trans to the bonded S atom. This trans bond lengthening effect through intermolecular contacts is also observed in the solid state for chlorides and organochlorides of sulfur and selenium and is discussed in greater detail for these compounds by Wynne.¹⁴

There are two notable similarities in the bond angles of SbCl_3dmit and AsCl_3dmit . The "linear" Cl-M-Cl bonded arrangements in both of these complexes are very close in bond angles. For AsCl_3dmit this angle is 168.7 (1)° as compared to 167.4 (1)° for Cl(3)-Sb-Cl(1) in SbCl_3dmit . What is more striking is the unusually large deviation from the ideal 90° angle observed for the S-M-Cl(ax) angle in both complexes where the chlorine atom noted is the atom involved in bridging. In SbCl_3dmit , S-Sb-Cl(3) is 76.2 (1)° as compared to the analogous angle in AsCl_3dmit of 77.9 (1)°. The other S-M-Cl(ax) angle in both cases does not show a great deviation. In SbCl_3dmit Cl(1)-Sb-S is 91.3 (1)° corresponding to Cl(2)-As-S of 91.2 (1)°. There are no other remarkable in-

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termolecular contacts other than those noted above for SbCl_3dmit .

The single-crystal X-ray study reported above thus provides further support for the solid-state vibrational data assignments reported earlier. As was the case for SeBr_2mtu and AsCl_3dmit , there was no clear evidence of bridging in the solid-state spectra of SbCl_3dmit .

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Supplementary Material Available: Listing of observed and calculated structure factors (5 pages). Ordering information is given on any current masthead page.

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Chemistry of Multisite-Bound Ligands: Crystal and Molecular Structure of $\text{HRu}_3(\text{CO})_8(\text{C}\equiv\text{C}-t\text{-Bu})(\text{PPh}_2\text{OEt})$

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One goal of our current research programs is to investigate the chemical reactivity associated with the multisite coordination of unsaturated ligands to several metal atoms in metal carbonyl clusters.^{2,3} In attempts to establish distinct patterns of reactivity for cluster-bound ligands, the polynuclear μ -acetylido compounds serve as particularly useful models since a progression of bonding modes, $\mu_2\text{-}\eta^2$,⁴ $\mu_3\text{-}\eta^2$,^{5,6} and $\mu_4\text{-}\eta^2$,⁷ is accessible, and furthermore the problem of ligand mobility frequently associated with other ligands such as CO is largely circumvented.⁸ In studies relating to the susceptibility of $\mu_3\text{-}\eta^2$ -acetylides to nucleophilic attack by uncharged ligands, a number of substitution products derived via carbonyl displacement from the neutral hydride $\text{HRu}_3(\text{CO})_9(\text{C}\equiv\text{C}-t\text{-Bu})$ ⁵ and the related "open" phosphide-bridged trimer $\text{Ru}_3(\text{CO})_9(\text{C}\equiv\text{CR})(\text{PPh}_2)(\text{R} = t\text{-Bu}, i\text{-Pr})$ ⁶ have been characterized. Current interest in such derivatives⁹ prompts us to report an accurate determination of the molecular structure of $\text{HRu}_3(\text{CO})_8(\text{C}\equiv\text{C}-t\text{-Bu})(\text{PPh}_2\text{OEt})$.

Table I. Crystal Data and Intensity Collection

formula	$\text{C}_{28}\text{H}_{25}\text{O}_9\text{PRu}_3$
mol wt	839.69
cryst system, space group	triclinic, $P\bar{1}$
cell	$a = 9.141(1)$, $b = 11.706(2)$, $c = 15.057(2)$ Å; $\alpha = 96.73(1)$, $\beta = 84.36(1)$, $\gamma = 100.31(1)^\circ$
Z	2
d_{measd}	1.78 g cm ⁻³
d_{calcd}	1.778 g cm ⁻³
V	1568.1(4) Å ³
F(000)	824
T	22 ± 1 °C
radiation	graphite-monochromated Mo K α ($\lambda = 0.71073$ Å)
diffractometer	Syntex P2 ₁
scan type	θ -2 θ
scan speed	variable (2-29.3° min ⁻¹)
scan width	[2 θ (Mo K α_1) - 0.8°] → [2 θ (Mo K α_2 + 0.8°)]
background	stationary counter-stationary crystal before and after each scan
reflectns measd	4136 ($2\theta < 45^\circ$)
reflectns obsd	3538 ($I \geq 3\sigma(I)$)
$\mu(\text{Mo K}\alpha)$	14.93 cm ⁻¹
R	0.024
R_w	0.028

Table II. Fractional Atomic Coordinates ($\times 10^4$) for $\text{HRu}_3(\text{CO})_8(\text{C}_2-t\text{-Bu})(\text{P}(\text{OEt})\text{Ph}_2)$

	x	y	z
Ru(1)	199.2 (4)	4239.5 (3)	3134.4 (2)
Ru(2)	688.8 (4)	1924.5 (3)	2672.4 (2)
Ru(3)	2912.5 (4)	3645.0 (3)	3468.3 (3)
P	1950.4 (12)	448.6 (9)	2078.5 (7)
O(1)	-1802 (4)	1108 (3)	1455 (3)
O(2)	-820 (5)	719 (4)	4296 (2)
O(3)	196 (6)	6790 (4)	3911 (3)
O(4)	-1769 (4)	4197 (4)	1612 (3)
O(5)	-2376 (5)	3151 (4)	4387 (3)
O(6)	5632 (4)	2670 (4)	2604 (4)
O(7)	2498 (5)	2124 (4)	5025 (3)
O(8)	4853 (6)	5873 (4)	4262 (3)
O(9)	3068 (3)	702 (3)	1200 (2)
C(1)	-875 (5)	1398 (4)	1931 (3)
C(2)	-256 (5)	1130 (4)	3680 (3)
C(3)	214 (6)	5859 (5)	3601 (4)
C(4)	-1050 (5)	4228 (4)	2198 (4)
C(5)	-1415 (6)	3532 (5)	3916 (4)
C(6)	4598 (6)	3017 (4)	2937 (4)
C(7)	2647 (6)	2670 (5)	4435 (4)
C(8)	4126 (7)	5057 (5)	3986 (4)
C(9)	1782 (4)	3351 (4)	2220 (3)
C(10)	2373 (4)	4471 (4)	2290 (3)
C(11)	3201 (5)	5388 (4)	1716 (3)
C(12)	4776 (6)	5163 (6)	1465 (5)
C(13)	3242 (9)	6612 (5)	2180 (4)
C(14)	2419 (7)	5250 (6)	853 (4)
C(15)	2791 (7)	1374 (5)	518 (4)
C(16)	4160 (10)	1570 (7)	-96 (5)
C(17)	758 (5)	-914 (4)	1699 (3)
C(18)	-424 (5)	-1412 (4)	2256 (4)
C(19)	-1322 (6)	-2448 (5)	1970 (4)
C(20)	-1086 (7)	-2978 (5)	1118 (4)
C(21)	77 (8)	-2497 (5)	560 (4)
C(22)	1010 (6)	-1462 (4)	841 (3)
C(23)	3215 (4)	-44 (4)	2752 (3)
C(24)	2700 (6)	-453 (4)	3571 (3)
C(25)	3654 (7)	-854 (5)	4074 (4)
C(26)	5101 (6)	-860 (5)	3776 (4)
C(27)	5636 (6)	-464 (5)	2962 (4)
C(28)	4701 (5)	-56 (4)	2439 (4)
H(1)	1383 (50)	4199 (39)	3988 (31)

Experimental Section

The synthesis of the title compound and a wide variety of substitution products will be described in detail elsewhere.¹⁰ Crystals

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